

VIA EFS**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In Re Patent Application of :

Takashi Ito, et al.

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Conf. No.: 8148

§ Group Art Unit: 1753

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Appln. No.: 10/562,098

§ Examiner: Anca Eoff

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Filing Date: December 21, 2005

§ Attorney Docket No.: 9369-114US
(T37-196236C)Title: ACTINIC RADIATION-CURABLE STEREOLITHOGRAPHIC RESIN
COMPOSITION HAVING IMPROVED STABILITY**DECLARATION OF TSUNEO HAGIWARA UNDER 37 C.F.R. § 1.132**

I, Tsuneo Hagiwara, declare and state as follows:

1. I am a co-inventor of the invention described and claimed in the above-identified patent application ("present application").

2. I received a Ph.D. in chemistry from the University of Tsukuba, Japan in March 1990.

3. Since April 2001, I have been employed by CMET Inc., an assignee of the present application. I have been engaged in the research and development of photoresins for stereolithography.

4. I am familiar with the above-referenced application, and in particular with the Office Action dated June 29, 2007 (Paper No. 20070611). I am submitting the present Declaration to overcome any § 102 rejections of claims 1-8 by demonstrating that the prior art does not teach or suggest sulfonium salts having formula (I) with 100% purity which contain less than 3% by mass of a compound of formula (II).

The Examiner's Position and the Purpose of the Declaration

5. The Examiner has taken the position that the closest prior to the invention includes U.S. Patent No. 5,783,358 of Schulthess ("Schulthess"), International Patent Publication Number WO 02/48101 ("WO '101"), using U.S. Patent Application Publication No. 2004/0030158 as an English equivalent), and U.S. Patent Application Publication No. 2004/0137368 of Steinmann ("Steinmann").

6. In the Office Action dated June 29, 2007, the Examiner rejected claims 1-6 under 35 U.S.C. § 102(b) as being anticipated by Schulthess or under 35 U.S.C. § 103(a) as being obvious over Schulthess in view of WO '101. The Examiner also rejected claims 1-8 under 35 U.S.C. § 102(e) as being anticipated by Steinmann or under 35 U.S.C. § 103(a) as being obvious over Steinmann in view of WO '101. The Examiner argued that Schulthess and Steinmann teach compositions containing, among other components, a sulfonium salt having formula (I) as a photo-initiator for cationic polymerization which the Examiner assumes has 100% purity.

Alternatively, the Examiner argues that it would have been obvious to utilize the sulfonium compound of WO '101 having 96% purity in the compositions of Schulthess or Steinmann.

7. The purpose of this Declaration is to demonstrate that the prior art sulfonium salts of Schulthess and Steinmann do not have 100% purity, as assumed by the Examiner. This demonstration will thus overcome the § 102 rejections of claims.

Background and Purpose of the Invention

8. The present invention is directed to an actinic radiation-curable resin composition for stereolithography which includes a cationic polymerizable organic compound, a radical polymerizable organic compound, a photo initiator for radical polymerization and a photo initiator for cationic polymerization which contains a highly pure (97 weight % or higher) compound represented by formula (I) and which contains less than 3% by mass of a compound

having formula (II). This compound is highly pure to improve the aging stability during operation and the storage stability of the composition.

9. It is known that compounds containing particular polymerization photoinitiators have poor stability. Therefore, as demonstrated in the Examples and the Comparative Examples of the present application, utilization of a highly pure sulfonium compound having formula (I) is critical to providing the observed stability properties. More specifically, the presence of a bissulfonium compound having formula (II) in the cationic polymerization initiator has a remarkable (undesirable) effect on the resulting properties.

10. As described in the present application and shown in Table 1, an inventive composition prepared in Examples 1 and 2 at pages 29 and 31 (using a cationic polymerization initiator containing a sulfonium compound of formula (I) having 98% purity) showed little increase in viscosity even after storage at a high temperature of 80°C for a long period of time (high storage stability). These resin compositions quickly cured molding due to actinic radiation irradiation to smoothly give an object by stereolithography having superior mechanical properties, appearance and color tone.

11. In contrast, in the comparative resin compositions, prepared in Comparative Examples 1 and 2 at pages 30 and 33, which utilized a commercial cationic polymerization initiator containing both a sulfonium compound having formula (I) and a bissulfonium compound having formula (II) in a mass ratio of 2:2, the viscosities began to increase after two days under heating at 80°C, and stirring became impossible after four days due to the excessively high viscosities. Accordingly, stereolithography could no longer be performed. These results demonstrate the criticality of the purity of the cationic polymerization initiator having formula (I), and the negative effects which are obtained by including significant amounts of a compound having formula (II). Accordingly, it is critical that the cationic polymerization initiator having formula (I) be at least 97% pure and include less than 3% by mass of a compound of formula (II).

Experimental Procedure and Results

12. Analysis of CIBTOOL™ SL 5180: A sample of CIBTOOL™ SL 5180, described in Example 1 of Schulthess, was commercially obtained. The sample was analyzed in the Analysis Laboratory of Teijin Limited using liquid chromatography and NMR to determine its chemical composition. As shown in Table A in Appendix A attached hereto, CIBTOOL™ SL 5180 was found to contain 2-3 weight % UVI 6974, a commercially available cationic polymerization initiator which is also taught in paragraph [0076] of Steinmann.

13. As shown in Row "E" of Table A, UVI 6974 contains two compounds, a monosulfonium salts (corresponding to a compound of formula (I)) and a bissulfonium salt (corresponding to a compound of formula (II)).

14. Attached hereto as Appendix B is the Material Safety Data Sheet (MSDS) for UVI 6974. As shown at page 2, UVI 6974 contains sulfonium (thiodi-4,1-phenylene)bis(diphenyl-bis[(OC-6-11)hexafluoroantimonate(1-)]) in an amount of up to 50% and p-thiophenoxyphenyldiphenylsulfonium hexafluoroantimonate in an amount of up to 50%: a compound of formula (I) in an amount of up to 50% and a compound of formula (II) in an amount of up to 50%. Therefore, CIBTOOL™ SL 5180 does not contain 100% of a sulfonium salt of formula (I).

Discussion and Conclusion

15. It can thus be seen that the compound used by Shulthess, CIBTOOL™ SL 5180, does not contain 100% of a compound of formula (I). Additionally, the cationic polymerization initiator taught by Steinmann, UVI 6974, also does not contain a compound of formula (I) of 100% purity.

16. This Declaration demonstrates that the commercially available CIBTOOL™ SL 5180 contains UVI 6974, a cationic polymerization initiator which contains up to 50% by weight of each of two sulfonium compounds, a monosulfonium compound having formula (I) and a bissulfonium compound having formula (II). UVI 6974 is a cationic polymerization initiator taught by both Schulthess and Steinmann, and thus both references teach the use of a material

which includes a compound of formula (II) and neither teaches a compound of formula (I) having 100% purity.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code; and that such willful statements may jeopardize the validity of the application or any patent issuing thereon.

Date: Nov. 22, 2007

Tsuneo Hagiwara

Tsuneo Hagiwara

Attachments: Appendix A: Table; Appendix B: MSDS

Table A.

APPENDIX A

Analysis of SL-5180		
Compound	Classification	Amount Wt%
A	Epoxy	~50
B	Epoxy	~16
C	Photo radical initiator	2~3
D	Triol	~1
E	Photo cationic initiator UVI-6974	2~3
F Low molecular aromatic compound	Sensitizer	Trace
G	Epoxy acrylate	(5)~10
H	Polyacrylate	3~5
I	Triol	5
J	Triacrylate	8



UNION CARBIDE CORPORATION
A Subsidiary of The Dow Chemical Company

MATERIAL SAFETY DATA SHEET



Product Name: CYRACURE(TM) PHOTOINITIATOR UVI-6974
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Union Carbide urges each customer or recipient of this MSDS to study it carefully to become aware of and understand the hazards associated with the product. The reader should consider consulting reference works or individuals who are experts in ventilation, toxicology, and fire prevention, as necessary or appropriate to use and understand the data contained in this MSDS.

To promote safe handling, each customer or recipient should: 1) Notify its employees, agents, contractors and others whom it knows or believes will use this material of the information in this MSDS and any other information regarding hazards or safety; 2) Furnish this same information to each of its customers for the product; and 3) Request its customers to notify their employees, customers, and other users of the product of this information.

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

1.1 IDENTIFICATION

Product Name	CYRACURE(TM) PHOTOINITIATOR UVI-6974
Chemical Name	Not applicable (mixture)
Chemical Family	Not applicable (mixture)
Formula	Not applicable (mixture)
Synonym	None

1.2 COMPANY IDENTIFICATION

Union Carbide Corporation
A Subsidiary of The Dow Chemical Company
39 Old Ridgebury Road
Danbury, CT 06817-0001

1.3 EMERGENCY TELEPHONE NUMBER

24 hours a day: CHEMTREC 1-800-424-9300.

Number for non-emergency questions concerning MSDS (732) 563-5522
Additional information on this product may be obtained by calling the Union
Carbide Corporation Customer Service Center at 1-800-568-4000.

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2. COMPOSITION INFORMATION

Component	CAS #	Amount (%W/W)
Sulfonium,(thiodi-4,1-phenylene) bis[diphenyl-bis[(OC-6- †1)hexafluoroantimonate(1-)]	89452-37-9	<= 50%
p-Thiophenoxyphenyldiphenylsulfonium hexafluoroantimonate	71449-78-0	<= 50%
Propylene carbonate	108-32-7	<= 50%

3. HAZARDS IDENTIFICATION

3.1 EMERGENCY OVERVIEW

Appearance Pale yellow to amber

Physical State Liquid

Odor Hydrocarbon

Hazards of product **WARNING!** CAUSES EYE IRRITATION.
MAY CAUSE ALLERGIC SKIN REACTION.

3.2 POTENTIAL HEALTH EFFECTS

Effects of Single Acute Overexposure

Inhalation Short-term harmful health effects are not expected from vapor generated at ambient temperature.

Eye Contact Injury to the cornea is not expected. Liquid causes irritation, experienced as stinging, excess blinking and tear production, with excess redness and swelling of the conjunctiva.

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Skin Contact Brief contact may cause slight irritation with itching and local redness.

Skin Absorption No evidence of harmful effects from available information.

Swallowing Low toxicity. May cause nausea.

Chronic, Prolonged or Repeated Overexposure

Effects of Repeated Overexposure Skin contact may cause sensitization and an allergic skin reaction.

Other Effects of Overexposure Curing of this product may release trace amounts of diphenyl sulfide, which has a nuisance odor. Overexposure to diphenyl sulfide may cause a subjective sensory response to the odor. Symptoms may include headache, nausea, light-headed sensation, abdominal discomfort or coughing. These symptoms do not typically reflect a toxic response to the product, and will generally self-resolve upon cessation of exposure.

Medical Conditions Aggravated by Exposure

A knowledge of the available toxicology information and of the physical and chemical properties of the material suggests that overexposure is unlikely to aggravate existing medical conditions.

3.3 POTENTIAL ENVIRONMENTAL EFFECTS

See Section 12 for Ecological Information.

4. FIRST AID PROCEDURES

4.1 INHALATION

Remove to fresh air.

4.2 EYE CONTACT

Immediately flush eyes with water and continue washing for several minutes. Remove contact lenses, if worn. Obtain medical attention if discomfort persists.

4.3 SKIN CONTACT

Remove contaminated clothing. Wash skin with soap and water. If irritation persists or if contact has been prolonged, obtain medical attention.

4.4 SWALLOWING

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If a large quantity (several ounces) has been swallowed, and if patient is fully conscious, give two glasses of water. Induce vomiting. This should be done only by medical or experienced first-aid personnel. Obtain medical attention.

4.5 NOTES TO PHYSICIAN

There is no specific antidote. Treatment of overexposure should be directed at the control of symptoms and the clinical condition of the patient.

Slight skin irritant.

Slight eye irritant.

Low toxicity by swallowing.

5. FIRE FIGHTING MEASURES

5.1 FLAMMABLE PROPERTIES

Flash Point - Closed Cup: *Pensky-Martens Closed Cup ASTM D 93* > 140 °C > 284 °F

Flash Point - Open Cup: *Not currently available.*

Autolignition Temperature: *Not currently available.*

Flammable Limits In Air:

Lower	2.3 % (V)
Upper	<i>Not determined.</i>

5.2 EXTINGUISHING MEDIA

Extinguish fires with water spray or apply alcohol-type or all-purpose-type foam by manufacturer's recommended techniques for large fires. Use carbon dioxide or dry chemical media for small fires.

5.3 EXTINGUISHING MEDIA TO AVOID

No information currently available.

5.4 SPECIAL FIRE FIGHTING PROCEDURES

No information currently available.

5.5 SPECIAL PROTECTIVE EQUIPMENT FOR FIREFIGHTERS

Use self-contained breathing apparatus and protective clothing.

5.6 UNUSUAL FIRE AND EXPLOSION HAZARDS

During a fire, hydrogen fluoride may be produced.

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See Section 8.3 - Engineering Controls

5.7 HAZARDOUS COMBUSTION PRODUCTS

May give off hydrogen fluoride gas if exposed to mineral acids or flame. Hydrogen fluoride can cause redness of skin and some burning and irritation of the nose and eyes at concentrations above 3 ppm in air (TLV = 3 ppm C).

6. ACCIDENTAL RELEASE MEASURES

Steps to be Taken if Material Is Released or Spilled:

Collect for disposal.

Personal Precautions: Wear suitable protective equipment. Avoid contact with eyes and skin.

See Section 8.2 - Personal Protection.

7. HANDLING AND STORAGE

7.1 HANDLING

General Handling

Avoid contact with eyes, skin, and clothing.

Keep container closed.

Use with adequate ventilation.

Wash thoroughly after handling.

FOR INDUSTRY USE ONLY.

Ventilation

General (mechanical) room ventilation is expected to be satisfactory.

7.2 STORAGE

Store in accordance with good industrial practices. Storage information may be obtained from product-specific Union Carbide Storage and Handling Guides, or by calling a Union Carbide Customer Service Representative.

8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

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8.1 EXPOSURE LIMITS

None established by OSHA, ACGIH or UCC.

8.2 PERSONAL PROTECTION

Respiratory Protection: Use self-contained breathing apparatus in high vapor concentrations.

Ventilation: General (mechanical) room ventilation is expected to be satisfactory.

Eye Protection: Safety glasses or monogoggles

Protective Gloves: Neoprene

Other Protective Equipment: Eye Bath, Safety Shower

8.3 ENGINEERING CONTROLS

PROCESS HAZARD: Sudden release of hot organic chemical vapor or mists from process equipment operating at elevated temperature and pressure, or sudden ingress of air into hot equipment under a vacuum, may result in ignitions without the presence of obvious ignition sources. Published "autoignition" or "ignition" temperature values cannot be treated as safe operating temperatures in chemical processes without analysis of the actual process conditions. Any use of this product in elevated-temperature processes should be thoroughly evaluated to establish and maintain safe operating conditions. Further information is available in a technical bulletin entitled "Ignition Hazards of Organic Chemical Vapor."

Trace amounts of benzene may be generated during ultraviolet (UV) curing of this material. The amount of benzene generated is dependent upon formulation parameters, process conditions and temperature. Benzene is a known human carcinogen and is included in the IARC, NTP and OSHA lists of carcinogens. Local ventilation is recommended for control of airborne vapor.

9. PHYSICAL AND CHEMICAL PROPERTIES

Physical State: Liquid

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Appearance: Pale yellow to amber

pH: *Not currently available.*

Solubility in Water (by weight): Insoluble

Odor: Hydrocarbon

Flash Point - Closed Cup: *Pensky-Martens Closed Cup ASTM D 93* > 140 °C > 284 °F

Percent Volatiles: ~ 50 Wt% Solvent

Boiling Point (760 mmHg): > 220 °C > 428 °F

Freezing Point: *Not determined.*

Specific Gravity (H₂O = 1): 1.4 25 °C / 25 °C

Vapor Pressure at 20°C: < 0.004 kPa < 0.03 mmHg

Vapor Density (air = 1): > 1

Evaporation Rate (Butyl Acetate = 1): < 0.01

Melting Point: *Not applicable.*

10. STABILITY AND REACTIVITY

10.1 STABILITY/INSTABILITY Stable

Conditions to Avoid: Contact with excessive heat, open flame, sparks, or ignition sources. Exposure to sunlight, or ultraviolet light. Fluorescent light.

Incompatible Materials: Mineral acids.

Hazardous Decomposition Products: May give off hydrogen fluoride gas if exposed to mineral acids or flame. Hydrogen fluoride can cause redness of skin and some burning and irritation of the nose and eyes at concentrations above 3 ppm in air (TLV = 3 ppm C).

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10.2 HAZARDOUS POLYMERIZATION Will Not Occur.

10.3 INHIBITORS/STABILIZERS Not applicable.

11. TOXICOLOGICAL INFORMATION

ACUTE TOXICITY

Peroral

Rat; female = 5000 mg/kg

Mortality: 0/5

Peroral

Rat; male = 5000 mg/kg

Mortality: 0/5

Percutaneous

Rabbit; female = 2000 mg/kg; 24 h occluded.

Mortality: 0/5

Percutaneous

Rabbit; male = 2000 mg/kg; 24 h occluded.

Mortality: 0/5

IRRITATION

Skin: Rabbit; 4 h occluded

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Results: minor erythema

Eye: Rabbit; 0.10 ml

Results: minor to moderate conjunctival irritation; no corneal injury

SENSITIZATION (ANIMAL AND HUMAN STUDIES)

Guinea pig, Intradermal injection of a 5% dilution in propylene glycol followed by topical application of a 50% dilution., Moderate to severe erythema in 12/20 upon challenge

SIGNIFICANT DATA WITH POSSIBLE RELEVANCE TO HUMANS

This material was mutagenic in the Ames bacterial assay. It is inactive, however, in the in vivo mouse micronucleus test.

12. ECOLOGICAL INFORMATION

12.1 ENVIRONMENTAL FATE

Information may be available, call Union Carbide.

12.2 ECOTOXICITY

Toxicity to Aquatic Invertebrates

Daphnia; 24 h; LC50

Result value: 4.4 mg/l

Toxicity to Aquatic Invertebrates

Daphnia; 48 h; LC50

Result value: 0.68 mg/l

12.3 FURTHER INFORMATION

None.

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13. DISPOSAL CONSIDERATIONS

13.1 WASTE DISPOSAL METHOD

Incinerate in a furnace where permitted under Federal, State, and local regulations. Dispose in accordance with all applicable Federal, State, and local environmental regulations. Empty containers should be recycled or disposed of through an approved waste management facility.

13.2 DISPOSAL CONSIDERATIONS

See Section 13.1

Disposal methods identified are for the product as sold. For proper disposal of used material, an assessment must be completed to determine the proper and permissible waste management options permissible under applicable rules, regulations and/or laws governing your location.

14. TRANSPORT INFORMATION

14.1 U.S. D.O.T.

NON-BULK

Proper Shipping Name : NOT REGULATED

BULK

Proper Shipping Name : NOT REGULATED

This information is not intended to convey all specific regulatory or operational requirements/information relating to this product. Additional transportation system information can be obtained through an authorized sales or customer service representative. It is the responsibility of the transporting organization to follow all applicable laws, regulations and rules relating to the transportation of the material.

15. REGULATORY INFORMATION

15.1 FEDERAL/NATIONAL

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COMPREHENSIVE ENVIRONMENTAL RESPONSE, COMPENSATION, AND LIABILITY ACT OF 1980 SECTION 103 (CERCLA)

The following components of this product are specifically listed as hazardous substances in 40 CFR 302.4 (unlisted hazardous substances are not identified) and are present at levels which could require reporting:

None.

SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT OF 1986 TITLE III (EPCRA) SECTIONS 302 AND 304

The following components of this product are listed as extremely hazardous substances in 40 CFR Part 355 and are present at levels which could require reporting and emergency planning:

None.

SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT OF 1986 TITLE III (EPCRA) SECTION 313

The following components of this product are listed as toxic chemicals in 40 CFR 372.65 and are present at levels which could require reporting and customer notification under Section 313 and 40 CFR Part 372:

Component	CAS #	Amount
Antimony compounds	Not available	<= 50.0000%

SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT OF 1986 TITLE III (EPCRA) SECTIONS 311 AND 312

Delayed (Chronic) Health Hazard : Yes
Fire Hazard : No
Immediate (Acute) Health Hazard : Yes
Reactive Hazard : No
Sudden Release of Pressure Hazard : No

TOXIC SUBSTANCES CONTROL ACT (TSCA)

All components of this product are on the TSCA Inventory or are exempt from TSCA Inventory requirements.

EUROPEAN INVENTORY OF EXISTING COMMERCIAL CHEMICAL SUBSTANCES (EINECS)

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This substance is listed on ELINCS., The number is 403-500-0.

CEPA - DOMESTIC SUBSTANCES LIST (DSL)

All components in this product are in compliance with the DSL.

15.2 STATE/LOCAL

PENNSYLVANIA (WORKER AND COMMUNITY RIGHT-TO-KNOW ACT)

This product is subject to the Worker and Community Right-to-Know Act. The following components of this product are at levels which could require identification in the MSDS:

Component	CAS #	Amount
Antimony compounds	Not available	<= 50.0000%

MASSACHUSETTS (HAZARDOUS SUBSTANCES DISCLOSURE BY EMPLOYERS)

The following components of this product appear on the Massachusetts Substance List and are present at levels which could require identification in the MSDS:

Component	CAS #	Amount
Benzene	71-43-2	<= 0.0010%

CALIFORNIA PROPOSITION 65 (SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT OF 1986)

This product contains the following chemical(s) known to the State of California to cause cancer and birth defects or other reproductive harm.

Component	CAS #	Amount
Benzene	71-43-2	<= 0.0010%

CALIFORNIA SCAQMD RULE 443.1 (SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT RULE 443.1, LABELING OF MATERIALS CONTAINING ORGANIC SOLVENTS)

VOC: Vapor pressure <0.03 mmHg @ 20°C

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699 g/l VOC

This section provides selected regulatory information on this product including its components. This is not intended to include all regulations. It is the responsibility of the user to know and comply with all applicable rules, regulations and laws relating to the product being used.

16. OTHER INFORMATION

16.1 AVAILABLE LITERATURE AND BROCHURES

ADDITIONAL INFORMATION: Additional product safety information on this product may be obtained by calling your Union Carbide Corporation Sales or Customer Service contact.

16.2 SPECIFIC HAZARD RATING SYSTEM

HMIS ratings for this product are: H - 2 F - 1 R - 0

NFPA ratings for this product are: H - 2 F - 1 R - 0

These ratings are part of specific hazard communications program(s) and should be disregarded where individuals are not trained in the use of these hazard rating systems. You should be familiar with the hazard communication applicable to your workplace.

16.3 RECOMMENDED USES AND RESTRICTIONS

FOR INDUSTRY USE ONLY

16.4 REVISION

Version: 3.1

Revision: 01/22/2001

Most recent revision(s) are noted by the bold, double bars in left-hand margin throughout this document.

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16.5 LEGEND

A	Asphyxiant
Bacterial/NA	Non Acclimated Bacteria
F	Fire
H	Health
HMIS	Hazardous Materials Information System
N/A	Not available
NFPA	National Fire Protection Association
O	Oxidizer
P	Peroxide Former
R	Reactivity
TS	Trade Secret
VOL/VOL	Volume/Volume
W	Water Reactive
W/W	Weight/Weight

The opinions expressed herein are those of qualified experts within Union Carbide. We believe that the information contained herein is current as of the date of this Material Safety Data Sheet. Since the use of this information and the conditions of the use of the product are not under the control of Union Carbide, it is the user's obligation to determine conditions of safe use of the product.